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(71) Applicant
Courtaulds PLC

(Incorporated in United Kingdom)

18 Hanover Square, London W1A 2BB

(72) Inventors
Roger Alan Chapman
Roger Bray
Paul John Akers

(74) Agent and/or Address for Service
J. Y. & G. W. Johnson,
Furnival House, 14–18 High Holborn, London WC1V 6DE

(54) Microwave-absorbing fibres and filaments

(57) Fibres or filaments adapted to absorb microwave radiation comprise synthetic fibres filled with soft ferrite, of general formula MFe_2O_r where M is a divalent metal cation, or iron particles, generally in amount at least 10% of the fibre volume. The soft ferrite or iron particles are dispersed in a fibre-forming polymer, preferably after treatment with a coupling agent such as a titanate ester, and the resulting dispersion is spun to form synthetic fibres. Preferred synthetic fibres are polyester, nylon and polypropylene.

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SPECIFICATION

Microwave-absorbing fibres and filaments

This invention is concerned with fibres and filaments adapted to absorb microwave radiation in the range of frbquencies 100 MHz to 300 GHz and, therefore, including radar frequencies of 600 MHz to 94 GHz.

According to the present invention a fibre or filament adapted to absorb microwave radiation comprises a synthetic fibre filled with soft ferrite or iron particles. The soft ferrite corresponds to the general formula M Fe_2O_4 where M is a divalent metal cation, e.g. Ni, Zn, Sr, Sb, Mn, Co, Mg, Cu or Cd. It also has a spinel crystal structure. We use the term "Synthetic Fibre" to include continuous filaments as well as staple fibres formed from such filaments.

The particle size of the iron or ferrite is generally in the range 0.1 to 100 microns and preferably 0.5–10 microns. Iron powder of this particle size is available commercially as "carbonyl iron". The iron or ferrite particles are preferably mainly spherical in shape.

The ability of the fibre to absorb microwave radiation increases with the ferrite and/or iron content which usually occupies at least 10 *per cent* of the volume of the fibre, for example 10–55 *per cent* and preferably between 15 and 35 *per cent*. It may be preferred to use both ferrite and iron as fillers to achieve absorption over a wider range of frequencies within the above range.

A process according to the invention for forming fibres or filaments adapted to absorb microwave radiation comprises dispersing soft ferrite or iron particles in a fibre-forming polymer and spinning the resulting dispersion to form synthetic fibres.

The filled synthetic fibre may be melt-spun, such as polyester, nylon and polyolefin, for example polypropylene. The filled synthetic fibre may alternatively be wet spun, for example viscose, certain acrylic fibres such as "Courtelle" (Registered Trade Mark of Courtaulds PLC) and alginate, or dry-spun, such as cellulose acetate or certain acrylic fibres, for example those spun from solution in dimethyl formamide.

In order to ensure a dispersion capable of trouble-free spinning, particularly at the higher loadings of iron and/or ferrite, it is preferred to pre-treat the iron and/or ferrite with from 0.1 *per cent* to 2 *per cent* by weight of a coupling agent which binds chemically with the surfaces of the iron or ferrite particles and additionally carries a plurality of organic radicals having an affinity for the fibre-forming substance. The pre-treated iron and ferrite particles have a reduced tendency to agglomerate, leading to a better dispersion in the fibre-forming substance. The iron and/or ferrite powder can be treated by adding the coupling agent in liquid form, optionally diluted, while the powder is agitated.

A useful family of commerically available coupling agents is based on titanate esters, preferably those conforming to the general formula:-

$$R_3\ Ti\ OR'$$

where each R, which may be the same or different, is a radical derived from an acidic organic compound, for example a carboxylic or a phosphoric acid, including a hydrocarbon chain (preferably an alkyl group) containing from 6 to 20 carbon atoms, and R' is an alkyl group, preferably containing 3 to 6 carbon atoms. A specific example, appropriate to iron or ferrite for dispersion in a fibre-forming polyolefin such as polypropylene, is isopropyl tri(dioctylpyrophosphato) titanate. Alternatives are isopropyl tri(dioctylphosphato) titanate and compounds in which R is an acyl group derived from a long chain fatty acid, e.g. a stearoyl or isostearoyl group, for example isopropyl triisostearoyl titanate.

A preferred embodiment of the invention is a polypropylene fibre filled with from 15 to 35 *per cent* by volume, preferably 15–30 *per cent*, of iron and/or soft ferrite pre-treated with a coupling agent. The preferred embodiment may be made by melt-spinning a homogenised blend of from 15 to 35 *per cent* by volume of iron and/or soft ferrite pre-treated with a coupling agent and from 85 to 65 *per cent* of a fibre-forming polypropylene, cooling the filaments so formed and optionally cold-drawing the filaments.

Such filaments may be used to make fabrics (woven, knitted or non-woven) to provide microwave absorption, the fabric being used in one or more plies to develop the required absorbing effect. Such fabrics or multiply fabrics may be used to make garments or screens to reduce the detection of individuals, vehicles or weapons by radar, drapes to separate areas, one of which contains a source of microwave radiation, or as a coating on some surfaces of a device generating microwave radiation.

The invention is illustrated by the following Examples:-

Example 1

750 grams of a nickel zinc soft ferrite of average particle size 2 to 6 microns was agitated in

a high speed mixer and 2.25 grams isopropyl tri(dioctylpyrophosphato) titanate was dripped in as a 1:1 mixture with mineral oil. 250 grams polypropylene powder was added and the mixture was fed to a compounding extruder, extruded as a tape and granulated. The granules obtained were fed to a melt spinning extruder, extruded with air cooling of the filaments formed and taken up as a 20 filament 2500 decitex yarn. The yarn contained 34 per cent by volume ferrite. The yarn was woven into a fabric which showed good absorption of radiation of radar frequencies.

Example 2

800 grams iron powder ("carbonyl iron" comprising spherical particles of average particle size 4 to 6 microns) was agitated in a high speed mixer. 2.4 grams isopropyl tri(dioctylpyrophosphato) titanate was dripped in as a 1:1 mixture with mineral oil and 200 grams polypropylene powder was added. This mixture was converted to a 20 filament 2200 decitex yarn by the procedure described in Example 1. The yarn contained 31 *per cent* by volume iron. The yarn was woven into a fabric which showed good absorption of radiation of radar frequencies.

Examples 3 to 5

Using the process of Example 1 20 filament yarn of 2000–25000 decitex were produced from the following mixtures:-

Example 3

Polypropylene containing 20 *per cent* by volume "carbonyl iron" powder as defined in Example 2.

Example 4

Polypropylene containing 35 *per cent* by volume "carbonyl iron" powder as defined in Example 2.

Example 5

Polypropylene containing 19 *per cent* by volume nickel zinc soft ferrite powder as defined in Example 1.

In each case the filler was pre-treated with 0.3 *per cent* by weight isopropyl tri(dioctylpyrophosphato) titanate.

The yarns of Examples 3 to 5 were woven into fabrics. The ability of the fabrics to absorb microwave radiation was measured by placing one or more layers of the fabrics in front of a metal plate of the same dimensions (20 × 18 cms). A radar beam was directed at the plate and the amount of radiation reflected back to the source was measured. The attenuation of reflected radiation compared to an uncovered metal plate was calculated. The results are shown in Table 1.

The reflectivity of the fabric in the absence of the metal plate was also measured and the reduction in reflectivity compared to the metal plate was calculated. The results are as shown in Table 2.

Table 1

Fabric of Example No.	*No. of layers of fabric*	*Frequency of radar*	*Attenuation*
3	1	18 GHz	2 dB
3	5	18 GHz	8 dB
4	1	18 GHz	1.5 dB
4	6	18 GHz	10 dB
5	1	12 GHz	0.4 dB
5	2	12 GHz	1.0 dB

Table 2

Fabric of Example No.	*No. of layers of fabric*	*Frequency of radar*	*Reduction in reflectivity*
3	1	7 GHz	15 dB
3	1	18 GHz	20 dB
5	1	7 GHz	20 dB
5	1	18 GHz	25 dB

CLAIMS

1. A fibre or filament adapted to absorb microwave radiation comprising a synthetic fibre filled with soft ferrite or iron particles.

2. A fibre or filament according to claim 1 in which the particle size of the iron or ferrite is in the range 0.5 to 10 microns.

3. A fibre or filament according to claim 1 or claim 2 which is filled with soft ferrite and iron particles.

4. A fibre or filament according to any of claims 1 to 3 in which the ferrite and/or iron content of the synthetic fibre is 10 to 55 *per cent* by volume.

5. A fibre or filament according to claim 4 in which the ferrite and/or iron content of the synthetic fibre is 15 to 35 *per cent* by volume.

6. A fibre or filament according to claim 1 in which the synthetic fibre is filled with soft ferrite in an amount of from 15 to 30 *per cent* by volume.

7. A fibre or filament according to any of claims 1 to 6 in which the synthetic fibre is a polyester, nylon or polyolefin fibre.

8. A fibre or filament according to claim 7 in which the synthetic fibre is polypropylene.

9. A process for forming fibres or filaments adapted to absorb microwave radiation comprising dispersing soft ferrite and/or iron particles in a fibre-forming polymer and spinning the resulting dispersion to form synthetic fibres.

10. A process according to claim 9 in which the fibre-forming polymer is thermoplastic and the filaments are melt spun.

11. A process according to claim 9 or claim 10 in which the iron and/or ferrite particles are treated with a coupling agent before they are dispersed in the fibre-forming polymer.

12. A process according to claim 11 in which the iron and/or ferrite particles are treated with 0.1 to 2 *per cent* by weight of the coupling agent.

13. A process according to claim 11 or claim 12 in which the coupling agent is a titanate ester.

14. A process for forming fibres or filaments adapted to absorb microwave radiation carried out substantially as herein described with reference to any of the Examples.

15. A woven, knitted or non-woven fabric adapted to absorb microwave radiation which is formed at least partly from fibres or filaments according to any of claims 1 to 8.

CLAIMS

Amendments to the claims have been filed, and have the following effect:-

Claims 1–3/6–15 above have been deleted or textually amended.

New or textually amended claims have been filed as follows:-

1. A fibre or filament adapted to absorb microwave radiation comprising a synthetic fibre filled with soft ferrite (as hereinbefore defined) and/or iron particles pre-treated with a coupling agent.

2. A fibre filament according to claim 1 in which the synthetic fibre is filled with soft ferrite particles (as hereinbefore defined).

3. A fibre or filament according to claim 1 or 2 in which the particle size of the ferrite and/or iron is in the range 0.5 to 10 microns.

6. A fibre or filament according to claim 2 in which the ferrite content of the synthetic fibre is 15 to 30 per cent by volume.

7. A fibre or filament according to any of claims 1 to 6 in which the coupling agent is a titanate ester.

8. A fibre or filament according to claim 7 in which the titanate ester has the general formula $R_3Ti\ OR'$, where each R independently is a radical derived from an acidic organic compound including a hydrocarbon chain containing from 6 to 20 carbon atoms and R' is an alkyl group.

9. A fibre or filament according to any of claims 1 to 8 in which the amount of coupling agent is 0.1 to 2 per cent based on the weight of ferrite and/or iron particles.

10. A fibre or filament according to any of claims 1 to 9 in which the synthetic fibre is a polyester, nylon or polyolefin fibre.

11. A fibre or filament according to claim 10 in which the synthetic fibre is polypropylene.

12. A process for forming fibres or filaments adapted to absorb microwave radiation comprising treating soft ferrite (as hereinbefore defined) and/or iron particles with a coupling agent, dispersing the treated ferrite and/or iron particles in a fibre-forming polymer and spinning the resulting dispersion to form synthetic fibres.

13. A process according to claim 12 in which the said particles are soft ferrite particles.

14. A process according to claim 12 or claim 13 in which the fibre-forming polymer is thermoplastic and the filaments are melt spun.

15. A process according to any of claims 12 to 14 in which the ferrite and/or iron particles are treated with 0.1 to 2 per cent by weight of the coupling agent.

16. A process according to any of claims 12 to 15 in which the coupling agent is a titanate ester.

17. A process for forming fibres or filaments adapted to absorb microwave radiation carried out substantially as herein described with reference to any of the examples.

18. A woven, knitted or non-woven fabric adapted to absorb microwave radiation which is

formed at least partly from fibres or filaments according to any of claims 1 to 11 or produced by a process according to any of claims 12 to 17.

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